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Characterisation of Bleached Hemp Pulps with the Use of the Computer Image Analysis Method

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Abstrac

Bleached kraft pulps made from 30 cm long hemp stalks, uncut hemp bast, and uncut hemp wood were prepared. Then the properties of these pulps and their fibres were analysed using the computer image analysis method. The results gave more exact knowledge of the fractional composition and fibre attributes of this raw material and also allowed its suitability for papermaking to be evaluated. Furthermore bleached kraft pulps from hemp stalks cut into 3 – 6 mm segments and hemp bast fibres cut into approx. 3 mm segments were analysed by the same method. The results obtained were analysed and compared with the attributes of bleached birch and pine pulps.

Key words: computer image analysis, industrial hemp pulps, composition of pulp, fibre attributes.

Introduction

Due to the stable increase in paper production, the shortage of a sufficient amount of fibrous raw material may be an essential issue for papermaking in the future. As found in literature, to solve this problem, it will be necessary to increase the amount of fibrous pulps obtained from wood growing in tree plantations and from waste paper. The wider utilisation of semi-manufactured products produced from plants obtained from agricultural cultivation is also taken into account. One such fibrous plant is industrial hemp (Cannabis sativa L.) [1 – 4].

Research into the use of industrial hemp has also been carried out in Poland [5-8], which mainly because the workers of the Institute of Natural Fibres have a long history of achievements in the cultivation of industrial hemp [9, 10]. In these studies, conditions for the pulping process of hemp raw materials (stalks, wood, and hemp bast fibres) using the kraft method [7], and the effects of the oxygen delignification and bleaching of the kraft pulps obtained were determined [8]. It is also essential to study more exactly the fibrous composition of industrial hemp pulps and the morphological properties of fibres of this plant. As reported in literature, this enables the properties of paper made from this kind of pulp to be better understood [11 - 15].

For decades, the determination of these kinds of properties has caused much difficulty because it was labour- and time-consuming [16, 17]. Nowadays, owing to the development of methods based on computer image analysis of fibrous slurry, it is possible to perform such analysis

in only a few minutes [18 – 21]. At least a few apparatuses that operate on the basis of this technology are available [22], one of which is the MorFi designed by Techpap. Using this apparatus, one can determine the following indices describing the fractional composition of pulps, the dimensions of fibres, and their degree of damage [23, 24]:

- the content of fibres in a mass unit of the pulp,
- the relative content of fines (fibres and elements of a length below 0.2 mm), which is calculated on the basis of the area of this fraction in a picture of fibrous slurry recorded by a digital camera
- the dimensions of fibres (length, width).
- the mass of a fibre length unit (coarseness),
- the percentage content of fibres which have kinks in the pulp,
- the average degree of fibre curl, calculated using the following equation: $curl = (1 I/L) \cdot 100$, where l is the highest fibre dimension and L its developed length.

In this paper we present the results of research performed in order to compare these properties of various bleached pulps made from industrial hemp raw materials (stalks, 'wood', and bast, fibres) as the one of the elements of the evaluation of their suitability for papermaking purposes.

Experimental

Pulp

Bleached pulps made from hemp raw materials were used in the study, of which

the common source was the stalks of industrial hemp (Cannabis sativa L.) of the Polish variety białobrzeska. The raw material was obtained from the Institute of Natural Fibres in Poznan. The pulps used in the research can be divided into two groups: The first group consisted of three pulps marked as Hs, Hb, and Hw, made from 30 cm long hemp stalks (considered and called uncut hemp stalk pulp in the text), uncut hemp bast separated manually from hemp stalks, and hemp wood (Hw), respectively. The second group of pulps consisted of two pulps marked as Hs3-6 and Hbf3, obtained from hemp stalks cut into 3 - 6 mm segments and hemp bast fibres cut into approximately 3 mm segments, respectively.

Hw, Hs, Hb, and Hs3-6 bleached pulps (brightness R457 nm: 87 – 91%) were obtained in our laboratory by the pulping of appropriate raw material using the kraft method and then subjecting the pulps obtained to oxygen delignification and bleaching with chlorine dioxide in conditions determined in earlier papers submitted for publication [7, 8].

For comparison, *Figure 5* (see page 98) and *Table 2* (see page 98) also include the results of computer image analysis of birch- and pine kraft pulp. These data are derived from experiments carried out as part of the authors' other research project [25].

Computer image analysis

The characterisation of the fibrous pulps by the computer analysis method was performed in MorFi apparatus according to the method described in the owner's manual for this apparatus [25].

Classical determination of fine contents

Ten grams of oven-dried pulp in 2 dm³ of water were disintegrated in a laboratory disintegrator for 3 min, after which it was transferred to a laboratory distributor and diluted with water to a total volume of 5 dm³. Then the suspension was poured into a Büchner funnel whose bottom had the form of a wire net with $75 \times 75 \mu m$ openings. Next the fraction of fibres was collected and diluted with water again to a total volume of 5 dm³, and finally it was filtered through the metal net once again. This procedure was repeated for each pulp until the water that passed through the metal net was visually clear. After the last cycle, the fraction of fibres was collected from the wire net of the Büchner funnel, dried, and weighed. Next, the amount of fines was calculated from the difference between the mass of the dry pulp sample subjected to analysis and the mass of the fibre fraction collected from the metal net after the end of the analysis.

Results and discussion

The characteristic feature of hemp stalks is their special anatomical structure (see *Figure 1*), in which apart from the inner woody layer (Hw), there is also an outer bast layer (Hb).

From the point of view of the use of hemp in papermaking, it is essential that these layers can be separated from each other, which gives the possibility of the separate processing of these two layers in order to obtain pulps with different papermaking properties. Moreover, it is possible to separate out bast fibres from the bast. Thus in the case of industrial hemp, a papermaking semi-manufactured product can be obtained from stalks, wood, bast, and bast fibres.

As reported in literature, the fibrous composition of these anatomical parts of hemp stalks and the dimensions of cells have been studied, but by a method based on observations under an optical microscope [2]. In our work, we characterised the fibrous composition of pulps from hemp wood, hemp bast, and also hemp stalks using modern computer image analysis, which enables a more exact determination of the properties of pulps

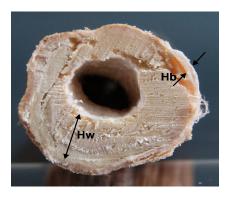


Figure 1. Cross-section of a hemp stalk; Hw - inner woody layer, Hb - outer last layer.

and their fibres for a considerably larger sample of pulp.

In the first part of the work, bleached pulps from wood of hemp stalks (Hw), uncut hemp bast (Hb) and uncut hemp stalks (Hs) were prepared and then subjected to examination using computer image analysis in the MorFi apparatus. It should be mentioned that the hemp wood pulp also contained pith cells, while the hemp bast pulp contained cuticle cells. These layers were connected to each oth-

er, and it would have been very difficult to separate them.

Results of the analysis of the Hw, Hb, and Hs pulps in the MorFi apparatus are presented in *Figure 2*.

The length distribution of fibres of the first pulp shows that hemp wood pulp (and therefore hemp wood) is composed of a large number of fibres of small length, ranging from 0.2 to 1.2 mm. This is confirmed by the image of hemp wood fibre suspension presented in *Figure 3.a*, recorded by the camera of the MorFi apparatus. As reported by Correia et al. [2], the hemp wood layer contains three essential cell types: tracheids, vessels, and parenchyma cells of 0.8 mm, 0.43 mm and 0.1 mm length, respectively. These elements are clearly visible in the photograph presented in Figure 3.b, taken with the optical microscope.

Secondly, hemp bast pulp was characterized using the computer image analysis method. This pulp was made from uncut hemp bast separated from hemp stalks by hand. As reported by Correia et al., in

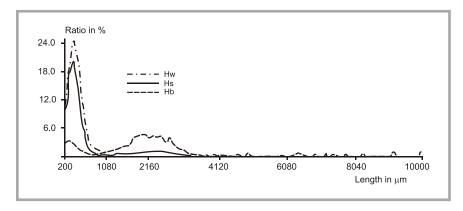
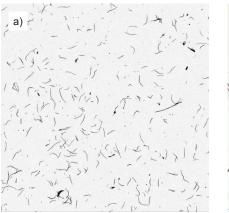


Figure 2. Distribution of fibre lengths of bleached pulps made from hemp wood (Hw), uncut hemp bast (Hb) and uncut hemp stalks.



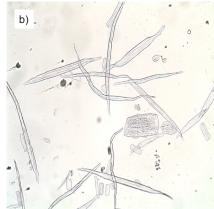


Figure 3. Image of the fibre suspension of hemp wood pulp a) recorded by the camera of the MorFi apparatus, b) taken under an optical microscope (magn. ×40).

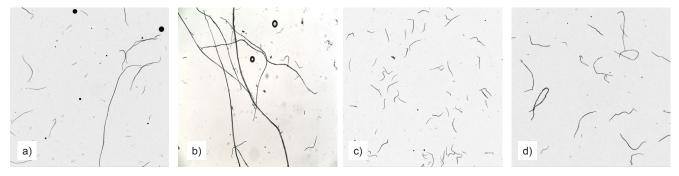


Figure 4. Images of fibre suspensions of (a, b) hemp bast pulp, (c) birch pulp, (d) pine-pulp, (a, c, d) registered by the camera of the MorFi apparatus, and (b) taken by an optical microscope (magn. ×40).

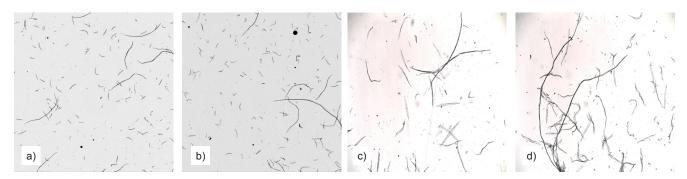


Figure 5. Image of the fibre suspension of hemp stalk pulp (Hs) (a, b) registered by the camera of the MorFi apparatus, and (c, d) taken by an optical microscope (magn. ×40).

studies in which pulp was obtained from the maceration of bast of a Canadian variety of hemp, the mean length of hemp bast fibres is 25 mm [2]. Apart from this, according to these authors, hemp bast also contains such cells as sieve tubes (mean length 0.8 mm) and parenchyma cells (mean length 0.35 mm).

Our research (Figure 2) shows that in the fibre length distribution of pulp made from hemp bast, several peaks of different magnitude are visible. As is known, the most characteristic feature of this anatomical part of hemp stalk is the presence of long elementary hemp bast fibres in it. It seems from Figure 2 that the fraction of these fibres is represented by several peaks corresponding to fibres of lengths in the range of 4 - 10 mm. Unfortunately, due to the MorFi apparatus, the fibre length can only be measured up to 10 mm, making it impossible to measure fibres longer than 10 mm in this pulp. However, the content of such fibres in the Hs pulp was determined by the method of directly counting single fibres. This revealed that elementary bast fibres longer than 10 mm constitute only about 1/5 of all bast fibres, while the fraction of fibres shorter than 10 mm is about 4/5 of all fibres. Thus, the characterisation of bast fibre pulp performed using computer image analysis showed the latter fraction of fibres above all.

The research performed enabled good characterisation of the fibrous composition of hemp bast pulp within a range of fibre lengths of 0.2 - 4.0 mm. *Figure 2* shows that apart from hemp bast fibres with a length of 4 - 10 mm, in hemp bast there are also many fibres of medium length (0.8 - 4.0 mm) and a number of short fibres with lengths in the range of 0.2 - 0.8 mm.

The presence of such short fibres confirms the images of the fibre suspension of Hb pulp presented in *Figure 4.a* and *4.b*, obtained using the MorFi apparatus and optical microscope, respectively. In order to show the similarity between the length of short fibres of hemp bast and fibres of typical papermaking pulps, images of bleached birch- and pine-pulp are presented in *Figure 4.c* and *4.b*.

The fibre length distribution of pulp made from 30 cm hemp stalks (Hs) was also analysed using the computer image analysis method. As *Figure 2* shows, it is characterised by two peaks, of which the greater corresponds to the fraction of fibres of lengths in the range of 0.2 - 0.8 mm (undoubtedly mainly of hemp wood), while the smaller corre-

sponds to the fraction of fibres of medium lengths, from 0.8 to 3.2 mm, in all probability deriving from hemp bast.

In the length distribution of hemp stalks pulp, however, peaks corresponding to the fraction of bast fibres of lengths from 4 to 10 mm, were not visible. This can be caused by their small number in the pulp of uncut hemp stalks, in which hemp wood fibres dominate, confirmed by the images of fibre suspensions of this pulp presented in *Figures 5.a, 5.b, 5.c* and *5.d.*

Apart from the fibre distribution, other properties of pulps and several properties of fibres can also be determined using computer image analysis, such as the content of fibres in a mass unit of pulp, the relative content of fines, and the mean length and width of statistical fibre, as well as its coarseness. Results of the determination of these indices are compiled in *Table 1*.

The data of *Table 1* show that hemp wood, hemp bast, and hemp stalks pulp contain 32.23, 2.90, and 16.52 mln of fibres per 1 gram of pulp, respectively.

As for the fines, the data of *Table 1* indicate that their relative content in the case of hemp wood and hemp stalks pulps amounted to 10 - 12% of the area

Table 1. Results of the analysis of bleached kraft pulps made from hemp wood (Hw), hemp bast (Hb), and hemp stalks (Hs) determined by the computer image analysis method using the MorFi apparatus; ¹ Determined using the classical method.

Index/unit	Type of pulp			
index/unit	Hw	Hb	Hs	
Length-weighted fibre length, mm	0.51	2.14	0.81	
Width, µm	23.7	20.6	23.2	
Coarseness, mg/m	0.067	0.269	0.118	
Fibres, mln/g	32.25	2.90	16.52	
Fine el. (% in area)	10.4	15.9	11.8	
Fines, wt%1	8.3	2.0	5.4	

of all elements, while in the pulp made of hemp bast it was as high as 16%. In order to verify the results obtained from the MorFi apparatus, the contents of fines were determined using the classical method (see the experimental section). The results are also presented in *Table 1*. These data show that the mass contents of fines in Hw and Hs pulp are 8.3 and 5.4%, respectively. Thus the results obtained from computer image analysis have higher values than those attained using the classical method. This was especially evident in the case of Hb pulp, whose mass content of fines was only 2 wt%. It seems that the cause of this difference may be the relatively low area of bast fibres, caused by their low number, which, in turn, results from the high coarseness of hemp bast fibres.

As for the fibre properties, the data of Table 1 indicate that the average length of fibres occurring in pulp made from hemp wood amounts to 0.51 mm, while in hemp bast pulp it is 2.14 mm. In both cases there are lengths lower than the those of 0.8 mm for hemp wood fibres and 25 mm for hemp bast fibres, as given by Correia et al. [2]. It seems that the our values of the mean length for hemp wood and hemp bast are closer to the real values of the mean length of hemp wood and hemp bast fibres because they were obtained on the basis of the measurement of the length of a considerably higher number of fibres than in the case of the authors mentioned.

Apart from the length, the statistical fibres of hemp wood and hemp bast also differ from each other in width and, especially, coarseness. As found from the data of *Table 1*, the coarseness of statistical hemp bast fibre is four times greater than that of statistical hemp wood fibre, which is confirmed by the image of these

fibres obtained using the optical microscope (*Figure 6*).

Table 1 also contains data concerning the properties of statistical fibre of pulp hemp stalks (Hs). The research found that such fibre shows medial values of length, width and coarseness in comparison with the values of these indices for fibres of hemp wood and hemp bast, which results from the fact that hemp wood and bast are the two main constituents of hemp stalks.

In the second part of the work, the morphological properties of bleached hemp pulp from hemp stalks cut into 3-6 mm segments (Hs3-6 pulp) and of pulp made from hemp bast fibres cut into ca 3 mm segments (Hbf3 pulp) were determined, analysed and compared with the same properties of hemp wood pulp (Hw), birch pulp (B) and pine pulp (P). The initial cutting of hemp stalks and bast seems to us to be an indispensable operation to prevent runability difficulties in industry, such as the entwining of these fibres around the elements of different machines (e.g. disintegrators) and the making of clews. The results of the research are presented in Figure 7 and Table 2

As shown in *Figure 7*, the fibre length distributions of Hs3-6 and Hbf3 pulps are

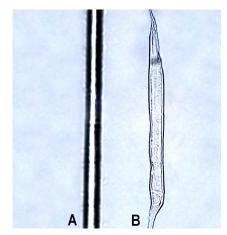


Figure 6. Fragment of hemp bast fibre (A) and hemp wood fibre (B). The photograph was taken under an optical microscope (magn. ×400).

characterised by similar shapes as in the case of Hs and Hb pulps (*Figure 2*). The only difference is the lack of a fibre bast fraction (4–10 mm) and fraction of short fibres in the fibre length distribution of Hbf3 pulp, which is caused by the operation of the initial cutting of hemp bast fibres and the fact that hemp bast fibres are separated from the tissue which accompanies them in the bast, which contains short fibres or relatively long parenchyma cells, as reported by Correia et al. [2].

In turn, comparing the fibre length distributions of Hs3-6 and Hw pulps with

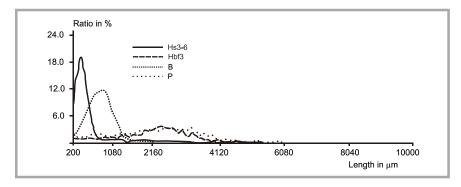
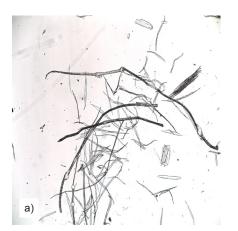


Figure 7. Fibre length distribution of bleached pulps produced from hemp stalks cut into 3-6 mm segments (Hs3-6) and bast fibres cut into 3 mm segments (Hbf3) against the background of the fibre length distribution of birch- and pine pulp (B and P respectively).

Table 2. Compilation of the results of the analysis of bleached kraft pulps made of hemp stalks cut into 3–6 mm segments (Hs3-6) and of the pulp obtained from hemp bast fibres cut into 3 mm segments (Hbf3); ¹ Determined using the classical method.

Index, unit	Hs3-6	Hbf3	Hw	В	Р
Length-weighted fibre length, mm	0.902	2.241	0.513	0.92	2.34
Width, µm	22.8	20.5	23.7	21.3	30.9
Coarseness, mg/m	0.111	0.258	0.067	0.117	0.192
Fibres, mln/g	16.98	2.64	32.25	11.07	3.84
Fine el. (% in area)	10.5	8.6	10.4	6.3	4.3
Fines, wt%1	5.7	1.0	8.3	2.7	2.2
Kinked fibres, %	28.1	37.7	30.5	53.4	56.5
Curl, %	6.9	12.6	7.2	11.0	15.3



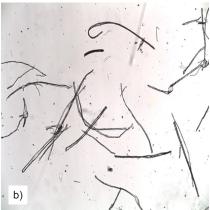


Figure 8. Image of the fibres suspension of bleached pulps made from (a) hemp stalks cut into 3-6 mm segments, and (b) hemp bast fibres cut into approx. 3 mm segments, both taken under an optical microscope (magn. $\times 40$).

those of pulps made of birch- and pinewood, one can state that hemp pulps contain considerably more short fibres than birch pulp. As for Hbf3 pulp, hemp pulp is characterised by a fibre length distribution close to that of pine pulp (P). Images of the fibres suspensions of Hs3-6 and Hbf3 pulps are presented in *Figure 8*.

Other properties of Hs3-6 and Hbf3 pulps and their fibres, which one can determine using the computer image analysis method, were also analysed and compiled in *Table 2*.

The data of *Table 2* show that the bleached pulp made of hemp stalks cut into 3 – 6 mm segments (Hs3-6) was characterised by average values of the fibre length, width and coarseness, and the content of fibres and fines was similar to the values of these indices for pulp made from uncut hemp stalks (Hs, *Table 1*). Essential differences can be observed in the values of the contents of fibres in 1 g of pulp and those of the content of fines in Hs3-6 and birch pulps, because in the case of Hs3-6 pulp these indices are considerably higher than for birch pulp.

Further analysis of the data in *Table 2* indicates that Hs3-6 pulp is characterised by average values of the fibre length, width and coarseness comparable with the values of these indices for birch pulp. However, it should be taken into account that the values of these indices for Hs3-6 pulp are influenced by their values for hemp wood and bast, which show essential differences in comparison with the values of these indices for birch- and pine pulp.

An analogical comparison with Hs3-6 pulp was also made for the pulp from hemp bast fibres separated and cut into 3 mm segments (Hbf3 pulp). As found from the data in *Table 2*, this pulp was characterised by values of the fibre length, width, coarseness, and fibre content in 1 g of pulp compared to the values of these indices determined for Hb pulp and presented in *Table 1*. The essential difference was in the content of fines in Hbf3 and Hb pulps, which in the case of Hbf3 pulp was lower by half.

The fibre indices of pulp made from hemp bast fibres and conventional pulps made from wood were also compared. As found from the data in Table 2, Hbf3 pulp was characterised by a fibre length comparable to that of pine pulp and by a fibre width comparable to that of birch pulp. In comparison to birch- and pine pulp, the coarseness of Hbf3 pulp was considerably higher (by 120 and 34%, respectively), indicating that statistical fibre of hemp bast fibre pulp has a higher cell wall thickness than the statistical fibre of pine pulp and, especially, birch pulp. Thus the fibre of hemp bast fibre pulp should be, in all probability, significantly stiffer than those of birch- and pine pulp.

The computer image analysis method also enables the determination of indices allowing evaluation of the degree of damage to pulp fibres. As found from the data in *Table 2*, the values of *kinked fibres* and *curl* indices of Hs3-6 and Hw pulps were lower than in the case of Hbf3 and birch- and pine-pulps. In all probability this is the result of the large content of short fibres in these pulps, which are less susceptible to transverse deformation.

Conclusions

- 1. The fibre length distribution of bleached pulp made from hemp wood showed that it contains only one fraction of fibres of lengths in the range of 0.2 - 1.2 mm, while in the case of pulp made of hemp bast, it contains as many as three fractions of fibres of lengths of 0.2 - 0.8 mm, 0.9 - 4 mm and 4 - 10 mm. As the fibre length that can be measured using MorFi apparatus is limited to 10 mm, it was not possible to determine the content of long (> 10 mm) fibres in hemp bast and hemp stalks pulps. The content of these fibres was, however, determined by the method of counting single fibres. The research showed that only in hemp bast pulp do 1/5 of all fibres have lengths greater than 10 mm.
- 2. The lengths of statistical fibres of hemp wood and bast amount to 0.513 mm and 2.138 mm, respectively. These values are lower than those given in literature on the subject. However, taking into consideration the high content of short fibres in hemp bast and relatively low content of fibres longer than 10 mm in it, the results obtained can be recognised as reliable.
- 3. Apart from the fibre length, the other property which distinguishes fibres of hemp bast and hemp wood is the coarseness, which for hemp bast fibres was four times greater than in the case of those of hemp wood. This can have a significant effect on the properties of paper made from hemp bast pulp and hemp wood pulp.
- 4. The fibre length distribution of pulp made from hemp stalks cut into 3 6 mm segments showed that it contains only two fractions of fibres of lengths in the ranges of 0.2 1.2 and 1.2 5.2 mm, while pulp made of hemp bast fibres cut into segments of approximately 3 mm contains only one fraction of fibres of lengths in the range of 0.2 mm to 4 mm. Thus, the initial cutting operation is an efficient way of shortening bast fibres that are too long for papermaking purposes.
- 5, Large differences were found in the number of fibres in 1 g of pulp made from hemp raw materials. The highest number of fibres in 1 g of pulp was found in hemp wood pulp (32 mln), followed by the pulp made of hemp stalks (17 mln), while the number was considerably lower in the case of hemp bast fibre pulp (2.6 mln). This

- can affect the optical properties of paper made from such semi-manufactured products.
- 6. The analysis of pulps made from hemp stalks and hemp wood using the computer analysis method and classical method indicates that they contain 2-3 times more fines than birch- and pine pulp. The lower content of fines was found in pulps from hemp bast and hemp bast fibres (1 - 2 wt%). In the case of these pulps, a relatively large difference between the results obtained using the computer image analysis method (content of fines expressed as a percentage of the area) and those found by the classical method (content of fines expressed in wt%) was found. It seems that the cause of this may be the relatively low area of specific hemp bast fibres, which results from their high coarseness.

Acknowledgments

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Multifilament Chitosan Yarn

The Institute of Bioploymers and Chemical Fibres is in possession of the know- how and equipment to start the production of continuous chitosan fibres on an extended lab scale. The Institute is highly experienced in the wet - spinning of polysaccharides, especially chitosan. The Fibres from Natural Polymers department, run by Dr Dariusz Wawro, has elaborated proprietary environmentlyfriendly method of producing continuous chitosan fibres with bobbins wound on in a form suitable for textile processing and medical application.



Multifilament chitosan yarn

We are ready, in cooperation with our customers, to conduct investigations aimed at the preparation of staple and continuous chitosan fibres tailored to specific needs in preparing non-woven and knit fabrics.

We presently offer a number of chitosan yarns with a variety of mechanical properties, and with single filaments in the range of 3.0 to 6.0 dtex.

The fibres offer new potential uses in medical products like dressing, implants and cell growth media.

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